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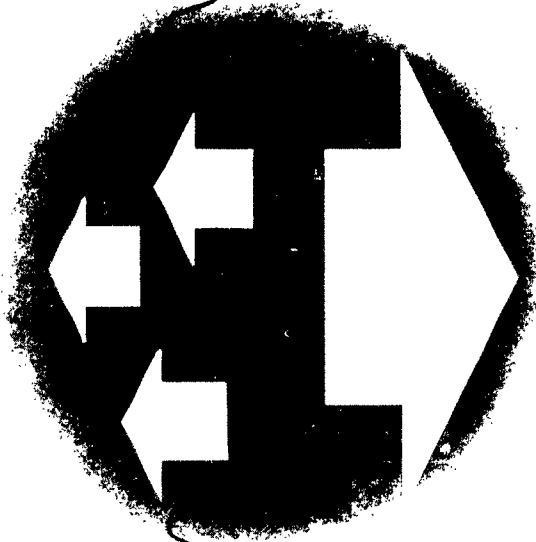
12 June 1952

Progress Report No. 820-28

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INVESTIGATION OF LIQUID ROCKET PROPELLANTS

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Contract N7onr-462

Task Order No. III

Project No. NR 220 023

Aerojet   A DIVISION OF

THE GENERAL TIRE & RUBBER COMPANY

THE GENERAL TIRE

12 June 1952   Progress Report No. 820-28

INVESTIGATION OF

LIQUID ROCKET PROPELLANTS

Contract N7onr-462
Task Order III
Project No. NR 220 023

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CONTRACT FULFILLMENT STATEMENT

This is the twenty-eighth in a series of progress reports submitted in partial fulfillment of Contract N7onr-462, Task Order III, covering the work performed in March and April 1952.

DEFINITION OF SYMBOLS

Symbol	Description	Units
C_F	Nozzle Coefficient	--
A_t	Nozzle Throat Area	$in.^2$
A_e	Nozzle Exit Area	$in.^2$
c^*	Characteristic Exhaust Velocity	ft/sec
I_{sp}	Specific Impulse	lb-sec/lb
F	Thrust	lb
p_c	Chamber Pressure	$lb/in.^2$
D_t	Nozzle Throat Diameter	in.
L^*	Characteristic Chamber Length	in.

I. INTRODUCTION

A.   Calculation of the performance of two methylhydrazines as rocket fuels shows that maximum specific-impulse values very close to that of hydrazine can be expected when these fuels are burned with oxygen. During this period laboratory investigations of the chemistry of these substituted hydrazines have been concerned with dehydration of methylhydrazine, the freezing temperatures of the hydrazine-methylhydrazine system, methylation of hydrazine, and a method of analysis of mixtures of possible reation products from the methylation of hydrazine.

B.   A study of the kinetics of the thermal decomposition of nitromethane at pressures above 200 psi is being made to obtain additional information about this monopropellant under conditions approximating those occurring in a rocket motor. A mass spectrometer is being used to follow the course of the decomposition of the fuel.

C.   Evaluation of the experimental performance of 13% solution of lithium borohydride in hydrazine prepared in this laboratory on a pilot plant scale has been completed. A maximum specific impulse of 256 lb-sec/lb, 94% of theoretical at this mixture ratio, was obtained.

II. STUDY OF HYDRAZINE DERIVATIVES

A.   INTRODUCTION

1.   The development of an ideal rocket fuel is necessarily a series of compromises among many desirable and undesirable features. Concentrated hydrazine possesses such superior qualities as high specific impulse and high density, but its freezing point is unacceptably high, and when burned with oxygen, the mixture ratio for optimum performance is such that 12% more hydrazine than oxygen by weight is required. Although the freezing point can be lowered by the use of additives, this invariably has resulted in lowered performance, increased viscosity, solid combustion products, or an increase in vapor pressure. Work to date in this laboratory has indicated that systems consisting of one or more of the methylhydrazines in hydrazine solution may overcome virtually all of the present disadvantages of hydrazine while preserving or even augmenting the desirable features. Thus the freezing points of such solutions meet Service requirements, and the mixtures possess theoretical performance values only negligibly less than that of hydrazine itself. Viscosity does not appear to be adversely affected, and the mixture ratio is shifted so that in the case of unsym.-dimethylhydrazine, 32% less fuel than oxygen is required to achieve optimum performance.

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2.   In order to evaluate a propellant properly, it is first necessary to investigate the properties of the pure material. Laboratory work has therefore been concentrated on the problems of the preparation and of the determination of the physical and chemical properties of two of the alkyl hydrazines: methylhydrazine and unsym.-dimethylhydrazine. Although the preparation of the latter compound in a pure state does not appear to be difficult, the former material is difficult to obtain in greater than approximately 95% purity. Concurrent with the study of the properties of these compounds, research is being conducted on new methods of direct methylation of hydrazine, in an attempt to prepare in a single step a mixture of subsitituted hydrazines with a low freezing point.

B.   METHYLHYDRAZINE

1.   Freezing Point - Composition Studies

a.   A sample of methylhydrazine analyzing 96.1% pure by potassium iodate oxidation was used for the determination of the freezing point vs composition curve for the hydrazine-methylhydrazine system, since considerable difficulty was encountered in preparing material of higher purity. The freezing point apparatus has been described before (Reference 1). The thermocouple now being used for temperature measurement is protected by a thin-walled glass tube instead of by Kel-F, as in the original design.

b.   The freezing point of the undiluted methylhydrazine was determined to be -56.08°C, a value 3.71° lower than that reported by Aston (Reference 2). Assuming Aston's value of the melting point to be correct, and taking his value of 2.4905 kcal/mole for the heat of fusion, the approximate mole fraction of methylhydrazine in the sample was estimated from the depression of the melting point, using the Schroder-Le Chatelier formula:

$$T_m = \frac{T_o}{1 - \dfrac{T_o R \ln x}{\Delta H_{fus}}}$$

where x is the mole fraction of the pure component with the melting point T_o. The mole fraction of methylhydrazine was calculated to be 0.907, and the weight fraction 0.961 if the impurity is assumed to be water, thus checking the analysis.

c.   On successively diluting the methylhydrazine with anhydrous hydrazine and determining the freezing temperature of the solutions increasingly rich in hydrazine, the eutectic for the system was found to lie at 85 wt% methylhydrazine, the temperature being -64°C (Figure 1). A plot showing mole% methylhydrazine vs freezing point and the theoretical curve is presented as Figure 2.

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        d.   The determination of the temperature corresponding to the appearance of first solids in solutions richer than approximately 50ᵤ in methylhydrazine was made difficult by the formation of glasses and by marked supercooling. The temperature at which last solids went into solution could be determined, however, by slowly warming a sample that had been completely frozen by a bath considerably below the expected freezing point. The viscosity of all solutions down to their freezing points did not appear to be excessive.

        2.   <u>Dehydration of Methylhydrazine</u>

        a.   Several dehydration procedures were tried in attempts to prepare 100% pure methylhydrazine. The most direct method for the liberation of the free base involves the neutralization of methylhydrazine sulfate with pellets of potassium hydroxide, according to the equation:

$$CH_3NHNH_2 \cdot H_2SO_4 + 2KOH = K_2SO_4 + CH_3NHNH_2 + 2H_2O.$$
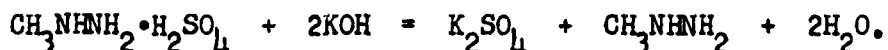
The product is distilled from the mixture and is then further treated with alkali to achieve greater purity. In one reaction, 0.5 mole of methylhydrazine sulfate was heated at $140^{\circ}C$ with potassium hydroxide, and the neutralization was initiated by the addition of a small quantity of water through a reflux condenser. The product was distilled at 30 to 50 mm Hg pressure. When 56 g of fused potassium hydroxide was added to combine with the water that distilled over, the solution separated into two phases: the upper layer (methylhydrazine) was a clear, light-brown liquid; the lower layer (aqueous potassium hydroxide) solidified on standing. The 20 ml of liquid was decanted and redistilled at reduced pressure to give a product of 92% purity. Lithium borohydride was then added to this material and the mixture was heated to $60^{\circ}C$ for 25 min in an attempt to remove chemically any water present, according to the reaction

$$2H_2O + LiBH_4 \longrightarrow LiBO_2 + 4H_2$$

The product was then distilled and the middle fraction was analyzed to be 95.6% methylhydrazine. Other methods of forming the free base, such as ammonolysis of the sulfate, are currently under investigation.

        b.   As Audrieth and Ogg (Reference 3) have pointed out, sodium hydroxide should be a more efficient dehydrating agent than potassium hydroxide; therefore, the method of liberating the free base was modified to involve the use of this compound. However, sodium hydroxide was found to possess several disadvantages which render its use impractical in the present case: (1) the neutralization reaction was very difficult to initiate; (2) the sodium sulfate formed during neutralization tenaciously retained the desired product; and (3) dehydration of an aqueous solution of methylhydrazine with fused sodium hydroxide occurred with separation into two phases, but additional fused alkali entered the methylhydrazine phase as a suspension. A satisfactory yield could be recovered from this reaction only by vacuum distillation to remove all volatile material, the addition of water to the solid reaction

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mass followed by distillation, and then the use of fused potassium hydroxide as a dehydrating agent in the combined distillates.

3. Volumetric Analysis of Methylhydrazine

After standing overnight, solutions of analyzed methylhydrazine appear to contain a component capable of oxidation by iodine monochloride, one of the end products in the iodate method of analysis. In one typical analysis, more iodate was added as iodine appeared after each apparent arrival at the end point, until, at the end of five weeks, the total quantity of standard iodate was 116% of the theoretical amount necessary to titrate the sample if it were all methylhydrazine. In an effort to determine whether some inherent side reaction made the analysis of methylhydrazine by iodate infeasible in contrast with information contained in References 4 and 5, a standard solution of methylhydrazine sulfate was prepared and analyzed under similar conditions. The initial end point was 100.0% of theoretical, and after 15 hr the amount of iodate corresponded to 102.0% of theoretical; the reaction seemed definitely to stop at this value. A titration performed with a calomel-platinum electrode system in the flask showed that the increase in potential and the disappearance of the iodine occurred simultaneously, thus also checking that the immediate visual end point gives an accurate method for the determination of the methylhydrazine content. Methyl alcohol, a possible product of oxidation, was unaffected by the conditions of the reaction, but a synthetic solution containing hydroxylamine, on the other hand, was slowly oxidized by an iodine monochloride solution in the manner observed in titrations of the free base. It thus appears at least possible that methylhydrazine prepared by the procedure described above contains small amounts of hydroxylamine or its methyl derivative, and that these compounds may have caused the anomalous results noticed in the analysis.

C. METHYLATION OF HYDRAZINE

1. Alkyl Halide Reactions

a. Attempts to methylate hydrazine by the action of methyl iodide and methyl bromide (Reference 1) at a mole ratio of hydrazine/methyl halide of 1.0 indicated that trimethyl azonium halide salts may have been the major product. Since the thermal decomposition of such a salt leads to a variety of products other than alkyl hydrazines (Reference 6), it was considered necessary to avoid its formation. This might conceivably be accomplished by using a large excess of hydrazine in the reactions, so that any mono- or di-substituted products would be less subject to further methylation, and by conducting the reactions at lower temperatures, in order to decrease the rate of conversion of the methylhydrazines to the azonium salt.

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b.   The action of methyl bromide on hydrazine in ethylene glycol solution (References 7 and 8) was investigated. Approximately 0.5 mole of methyl bromide was added dropwise to one mole of a rapidly stirred solution of hydrazine in 250 ml of the glycol at room temperature. The addition required 2 hr, and the resulting solution was clear and colorless. Ethylene glycol was considered a suitable solvent for this reaction because of its high boiling point (195°C), which should allow the easy removal of the more volatile methyl derivatives of hydrazine by distillation. The reaction mixture was treated with excess potassium hydroxide, refluxed for 3 hr, and the product was distilled. The fraction boiling in the range of 87 to 116°C showed a molecular weight of 70[*] by titration with a standard solution of potassium iodate, and thus contained an appreciable concentration of inert materials, the possibility of high molecular weight components being small. The product has been stored under a nitrogen atmosphere pending further analysis.

c.   As another means of preparing methylhydrazine and unsym.-dimethylhydrazine, the reaction of gaseous methyl bromide with refluxing hydrazine in the presence of solid potassium hydroxide was investigated. It was believed that by conducting the reaction under these experimental conditions, any methyl derivatives formed could be removed immediately from the reaction vessel by virtue of their greater volatility. The assumption was made that any salt formed (e.g. methylhydrazine hydrobromide) would be continuously neutralized by the potassium hydroxide present. The apparatus was so arranged that the hydrazine employed was refluxing at approximately three-fourths of the length of an air condenser at the time of addition of the methyl bromide. The latter compound was bubbled into the hot solution, and soon after the initial addition, vapors succeeded in traversing the entire length of the condenser; these were condensed and collected. A bubbler in the system indicated that no methyl bromide was lost by volatilization before it could react. The material collected by condensation amounted to only 5 ml (bp 107 to 110°C), which was disappointingly small, inasmuch as 70 g of methyl bromide was added to 150 g of hydrazine. The product was analyzed by iodate titration and showed a hydrazine content of 96%; precipitation as the picrate also indicated that the material was unsubstituted hydrazine, probably distilled over by the exothermic reaction. The material remaining in the reaction kettle has been stored over fused potassium hydroxide pending further analysis.

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[*]The molecular weights of hydrazine, methylhydrazine, and unsym.-dimethylhydrazine are, respectively, 32, 46, and 60.

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d.   In a study of the effect of lower reaction temperature, 0.5 mole of methyl bromide was added dropwise over a period of 2 hr to 1.0 mole of 96% hydrazine at 0°C. A white solid separated after approximately 0.25 mole of the bromide had been added. The dried solid product of the reaction produced an alkaline solution in water; hence it appears that both the temperature and mole ratio employed may have been inadequate in preventing the formation of the azonium salt.

2.   Dimethyl Sulfate Reactions

The low cost of dimethyl sulfate makes it an attractive methylating agent for hydrazine. Although previous procedures for the preparation of methylhydrazine have involved treatment of the hydrazine with benzaldehyde to form benzalazine before addition of methyl sulfate (Reference 9), the direct alkylation of hydrazine was attempted because of the possibility of eliminating one step from the synthesis. In a preliminary, small-scale reaction, the addition of a small quantity of methyl sulfate to 96% hydrazine produced an immediate, vigorous reaction. Successive additions were made until a solid, insoluble in an excess of dimethyl sulfate, appeared. A picrate of melting point 165°C (methylhydrazine picrate mp 166°C) was prepared from an aqueous solution of this solid. Because of the apparent success of the direct reaction, molar quantities of hydrazine and dimethyl sulfate were next allowed to react in five moles of absolute methanol at 0°C. Although initially cloudy, the solution was clear after standing for 15 hr, the small quantity of solids present having settled. A 25-ml aliquot was removed from the reaction vessel, neutralized, distilled, and then treated with sulfuric acid. This treatment yielded a solid (approximately 3 g) melting at 139°C (methylhydrazine sulfate mp 141°C). The entire reaction mixture was then treated in this manner to recover the product as the sulfate. The sulfate was neutralized with potassium hydroxide and the free bases were distilled and collected; material was collected continuously from 82 to 116°C, but no definite fraction was collected at the boiling point of methylhydrazine, indicating that techniques other than a simple distillation would have to be used to separate the various products.

D.   ANALYSIS OF MIXTURES OF METHYLHYDRAZINES

1.   It is at once apparent that in order to determine the constituents of many of the reaction products discussed above, an analytical procedure is required which is effective in distinguishing between hydrazine and its methyl derivatives. Moreover, since the odors of ammonia and methylamine were frequently noticed in the reaction products, such a method must be applicable in the presence of these materials, unless they can be eliminated by a suitable means, such as distillation. The presence of water in a sample could be held to a minimum by extended drying over fused potassium hydroxide.
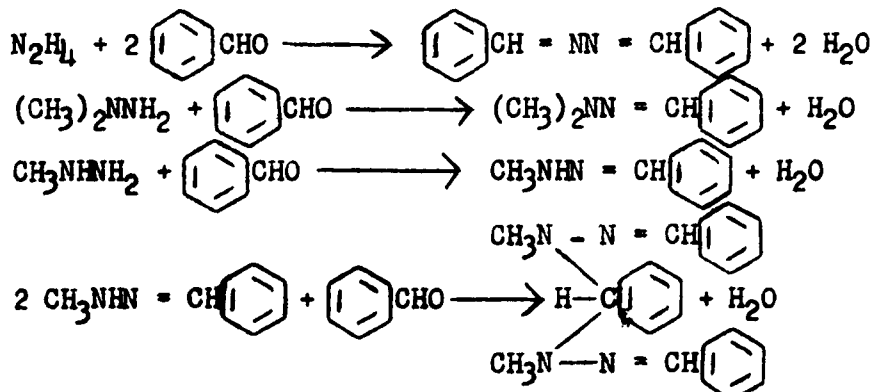
2. The use of ultraviolet absorption spectroscopy was considered in connection with the development of a suitable analytical technique. The use of p-dimethylaminobenzaldehyde as a complexing agent for hydrazine and its methyl derivatives proved unsuitable, since the complexes were formed only in acid solutions, and the complexing agent itself produces a yellow color in such solutions, to an extent dependent on the pH. A somewhat simpler system consisted of solutions of hydrazine and its derivatives in benzaldehyde. The choice of benzaldehyde as the solvent for the ultraviolet work proved unsuccessful, however; benzaldehyde itself absorbed strongly, and complicating solution effects caused inaccurate results when the determination of a synthetic mixture of hydrazine and methylhydrazine was attempted:

Actual Concentrations:	N_2H_4	CH_3NHNH_2
	0.0226g/ml	0.0255g/ml
Found:	0.0381	0.0248

3. The use of infrared spectroscopy was considered in connection with the analytical problem, but because of the highly polar character of hydrazine it was not feasible to employ sodium chloride cell windows, and the method was accordingly not reduced to practice.

4. A chemical method of analysis was therefore adopted. The procedure is based on the fact that hydrazine derivatives form complexes with benzaldehyde which differ in kind, and hence are more easily separated than are the parent substances. In the presence of an excess of benzaldehyde, hydrazine is converted to benzalazine (Reference 10), dimethylhydrazine to the hydrazone (Reference 11), and methylhydrazine to a product which may be regarded as a condensation of the initially formed hydrazone with additional benzaldehyde (Reference 7):

$$N_2H_4 + 2 \, C_6H_5CHO \longrightarrow C_6H_5CH = NN = CH\,C_6H_5 + 2 \, H_2O$$

$$(CH_3)_2NNH_2 + C_6H_5CHO \longrightarrow (CH_3)_2NN = CH\,C_6H_5 + H_2O$$

$$CH_3NHNH_2 + C_6H_5CHO \longrightarrow CH_3NHN = CH\,C_6H_5 + H_2O$$

$$2 \, CH_3NHN = CH\,C_6H_5 + C_6H_5CHO \longrightarrow \begin{array}{c} CH_3N - N = CH\,C_6H_5 \\ H - C\,C_6H_5 + H_2O \\ CH_3N - N = CH\,C_6H_5 \end{array}$$

The analytical procedure permits the separation of hydrazine and unsym.-dimethylhydrazine from a mixture of the three components; methylhydrazine may then be obtained by difference. In analyzing a mixture of reaction products from an attempted alkylation of hydrazine, care must be exercised to remove

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completely any water present by prolonged drying over fused potassium hydroxide. Any amines must be removed by volatilization under reduced pressure. When these impurities have been removed, the following procedure is applicable:

a.   A weighed amount of the solution to be analyzed (0.5 to 1.0 g) is added to a solution of an excess of benzaldehyde in 8 ml of ethanol. An excess of benzaldehyde will have been added if, in the calculation of the required amount of this reagent, the unknown solution is considered to consist entirely of hydrazine. The resulting yellow solution is heated to boiling, and water is added until the cloud point is reached. The solution is then clarified by the addition of ethanol. On cooling, benzalazine separates quantitatively (99.2%). The solution is filtered, the solid washed with 2 ml of a 3:1 water-ethanol mixture, and then vacuum-desiccated to constant weight. A refinement in the determination of hydrazine consists in applying a correction from the following table for the solubility of benzalazine in the particular water-ethanol solution employed in its precipitation.

Volume Ratio, $EtOH/H_2O$	Solubility, g/ml
0	0.0010
0.3	0.0010
1	0.0022
3	0.0104
infinite	0.0370

The filtrate from the benzalazine precipitation is used for the determination of <u>unsym.</u>-dimethylhydrazine. Water is added to the filtrate to cause the separation of an organic layer, the layers are separated, and the aqueous layer is extracted with benzene. An amount of picric acid which is sufficient to react with the undetermined portion of the sample if it consists entirely of <u>unsym.</u>-dimethylhydrazine is dissolved in the minimum quantity of boiling benzene, and this is added to the combined benzene extract and organic layer. The picrate of the hydrazone of <u>unsym.</u>-dimethylhydrazine separates nearly quantitatively (98%), and this is vacuum-desiccated and dried. In addition to a solubility correction, it is necessary to correct further for any benzalazine picrate, since this material is also insoluble in benzene. The following data are used for these corrections:

Compound	Solubility in Benzene at Room Temperature, g/ml
Benzaldehyde <u>unsym.</u>-dimethyl hydrazone picrate	0.0046
Benzalazine picrate	0.0240
Picric acid	0.1440

Methylhydrazine can then be obtained by difference.

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b.   The following results were obtained for a mixture of hydrazine, methylhydrazine, and <u>unsym.-dimethylhydrazine</u>:

Component	Wt of Component (g)	Wt% of Component (Calc)	Wt% of Component (Found)	Difference (%)
N_2H_4	0.294	57.4	58.7	+2.2
CH_3NHNH_2	0.114	20.3	19.2[*]	
$(CH_3)_2NNH_2$	0.104	22.3	22.1	-1.0
	0.512	100.0	100.0	

5.   In order to determine the methylhydrazine content of an unknown sample in a manner other than by difference from the total weighed amount of sample, it has been found that it is possible to obtain a value for total hydrazines by potentiometric titration. By following an iodate titration with a calomel-platinum electrode system, a rapid change in potential is found at the volume of iodate corresponding to a four-electron oxidation of hydrazine and methylhydrazine and a two-electron oxidation of dimethylhydrazine. After figures from the gravimetric analysis for hydrazine and dimethylhydrazine are obtained, it is then possible to calculate directly the amount of methylhydrazine present in the sample. Although this method is in a sense still by difference, it eliminates the possibility of including such inert materials as water in the figure reported for methylhydrazine as is allowed by the method reported above.

E.   CALCULATION OF THEORETICAL PERFORMANCE OF METHYLHYDRAZINES
AS FUELS WITH LIQUID OXYGEN

1.   Theoretical values of performance parameters for the combustion of methylhydrazine and <u>unsym.-dimethylhydrazine</u> with liquid oxygen in an ideal rocket engine at 300-psia chamber pressure were calculated on the basis of equilibrium composition during the expansion process for a range of fuel-oxidant ratios. The maximum value of specific impulse for the methylhydrazine and oxygen system is 272 lb-sec/lb and occurs at a mixture ratio of 1.23, and the maximum value for the N,N-dimethylhydrazine and oxygen system is 269 lb-sec/lb at a mixture ratio of 1.48. The other parameters included in the calculations were combustion-chamber temperature, nozzle-exit temperature, equilibrium gas composition, characteristic velocity, coefficient of thrust, and mean specific heat ratio of the gases between chamber and nozzle-exit conditions.

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[*]By difference.

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2.   The values of specific impulse for the methylhydrazine and oxygen system over a range of mixture ratios 1.04 to 1.74 are shown in Figure 3. The values for the N,N-dimethylhydrazine and oxygen system over the range of mixture ratios 1.17 to 1.71 are shown in Figure 4. The values of characteristic velocity, coefficient of thrust, and mean specific heat ratio for the respective systems are shown in Figures 5 and 6.

3.   The calculations were carried out by means of the method of Kandiner and Brinkley (Reference 12) using the Newton-Raphson modification of this method wherever necessary. In addition, the following customary assumptions were made: adiabatic combustion at constant pressure of 300 psia, isentropic expansion to one atmosphere, no friction, and homogeneous mixing. The products of combustion were assumed to be ideal gases and included the following substances: carbon monoxide, hydrogen, water, nitrogen, carbon dioxide, hydroxyl radical, nitric oxide, oxygen, atomic hydrogen, atomic nitrogen, and atomic oxygen.

4.   The thermodynamic data used for the combustion gases were based on those published by the National Bureau of Standards (Reference 13). The heats of formation of liquid methylhydrazine and N,N-dimethylhydrazine were based on the respective heats of combustion, 311.711 kcal/mole and 472.648 kcal/mole, as determined by Aston and co-workers (Reference 14). The resulting standard heats of formation are +12.69 kcal/mole and +11.27 kcal/mole for methylhydrazine and N,N-dimethylhydrazine, respectively.

5.   From the preliminary value of 473.5 kcal/mole (Reference 14), for the heat of combustion of liquid N,N'-dimethylhydrazine, it is estimated that this compound would have a specific impulse approximately 1 lb-sec/lb higher than the N,N-dimethylhydrazine when oxidized under the same conditions.

6.   Although the theoretical specific impulse of each of the methyl-substituted hydrazines is slightly less than that of hydrazine itself when liquid oxygen is the oxidant, there are certain other factors which are relatively advantageous from the standpoint of using these alkyl hydrazines as propellants. In addition to the more favorable freezing points (compared with hydrazine), there is an important factor of cost.

a.   Although it is not possible at this time to provide a reliable estimate of the ultimate cost of producing these alkyl hydrazines, it is probably safe to assume that when produced on a relatively small scale they will cost slightly more than hydrazine itself. Without knowing these costs, it is still possible to establish an upper limit below which these compounds would hold an advantage in comparison with hydrazine.

b.   By taking the present market prices for hydrazine and liquid oxygen, a base cost for a pound of propellant can be calculated, and from this the maximum permissible cost of the methylhydrazines which will make them just competitive is computed. This is the first item in column 4 of the following table:

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Compound	Mixture Ratio for Optimum Performance	Weight of Fuel per lb of Total Propellant for Optimum Performance	Cost of 1 lb of Fuel, Oxygen, and Total Propellant, $			Cost of Fuel / Cost of N_2H_4
			Fuel	Oxygen	Propellant	
N_2H_4	0.89	0.528	4.75	0.05	2.53	--
CH_3NHNH_2	1.23	0.448	5.57	0.05	2.53	1.17
$(CH_3)_2NNH_2$	1.48	0.403	6.20	0.05	2.53	1.31

Thus the two substituted hydrazines would have a theoretical cost advantage over hydrazine if the cost of production were to be not more than 117% of that of hydrazine for the mono-substituted compound, and not more than 131% for the di-substituted compound.

III. KINETIC STUDY OF THE THERMAL DECOMPOSITION OF NITROMETHANE

A.   INTRODUCTION

Preliminary experiments have been described (Reference 1) which show that the thermal decomposition of nitromethane at pressures above 200 psi can be studied conveniently in the temperature range of 300 to 350°C by employing glass ampoules to contain the samples. By analyzing the contents of ampoules heated for varied lengths of time by means of a mass spectrometer, it is believed that valuable information related to the mechanism of decomposition may be obtained under conditions more closely approaching actual rocket-motor conditions than have been used in previous investigations.

B.   APPARATUS AND PROCEDURE

1.   Views of the completed apparatus are shown in Figures 7 through 9. Although initially some difficulty was encountered because of the tendency of the "Blue Temp" heating bath (a mixture of sodium nitrate and nitrite) to "creep" up on the wall of the furnace core and short out the heating coils, this problem has been eliminated by enclosing the inner core of the furnace in an aluminum shell with a rim projecting above the furnace core.

2.   The following steps are taken in performing a thermal decomposition test:

a.   An ampoule made of 12-mm Pyrex tubing is cleaned with nitric acid, rinsed with distilled water, and dried. After an accurate determination of its volume (approximately 3 ml), the ampoule is filled with the desired amount of nitromethane and sealed off under vacuum. The weight

III Kinetic Study of the Thermal
   Decomposition of Nitromethane, B (cont.)

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of nitromethane is determined accurately by weighing the ampoule before and after sealing it. A simple gas-law calculation then gives the pressure that the nitromethane will initially exert when completely vaporized at the temperature of the furnace.

      b.   The ampoule is now placed inside the pre-heated metal holder (shown in Figure 8) and lowered quickly into the heating bath by means of a hydraulic jack and remote-control handles. The temperature of the bath is determined frequently by a chromel-alumel thermocouple located in the center of the furnace; the maximum temperature variation of the furnace during a test is usually less than $\pm 0.25^\circ C$. At the completion of the run, the ampoule is lifted out of the furnace and transferred to the quenching bath (water at room temperature) within 20 sec.

      c.   The ampoules containing nitromethane and its decomposition products are kept in dry ice until a mass spectrometric analysis can be made. At that time the contents of the ampoule are transferred to a 1000-ml flask by means of a tube-opener (Reference 15). Because of the presence of 20 to 40 mm of noncondensible gas pressure, small droplets of nitromethane may remain on the walls of the breaker and connecting tubing. Since their evaporation and diffusion into the flask is extremely slow, several experiments were carried out with ampoules containing known quantities of nitromethane and carbon dioxide to determine whether complete recovery can be effected after breaking the ampoule. It was found that by attaching the tube-opener directly to the flask, by pointing the break-off end of the ampoule into the flask, and by careful heating of the droplets, the loss can be brought within the limit of experimental error established by other factors.

      3.   Since the temperature measurements during the decomposition experiments are made by means of a thermocouple situated in the center of the furnace (Figure 7), it was necessary to determine whether the temperature measured by this couple is identical with the temperature at the location of the ampoule. Several measurements were therefore made in both positions. The experimental setup is shown in Figure 10. After allowing sufficient time to establish equilibrium, readings were taken alternately on the two thermocouples. On the basis of these tests, it was determined that the temperature measured in the center of the furnace is $0.14^\circ C$ below the value at the ampoule position. This correction is applied to all temperature measurements.

      4.   In order to ascertain the zero-time of the reaction it is necessary to know the time interval required to heat the ampoule to the furnace temperature. This time interval was determined by introducing into the furnace the ampoule and thermocouple shown in Figure 10, and taking temperature readings until equilibrium was obtained. The results of several tests show that approximately 2.7 min is required to reach the furnace temperature. The zero-time of the reaction is therefore taken as 2.7 min after introduction of the ampoule into the furnace. By determining the extent of the reaction during this initial period a correction can be applied to the results of the decomposition tests.

III Kinetic Study of the Thermal
    Decomposition of Nitromethane (cont.)

C. ANALYSIS

1. A satisfactory analytical procedure has not yet been worked out for the determination of the extent of decomposition. From the traces produced by the mass spectrometer, the quantity of nitromethane present in a mixture can be determined from a knowledge of the relationship of nitromethane pressure to the height of mass peak 61, the parent peak of nitromethane. This relationship must be ascertained both for pure nitromethane and for mixtures of nitromethane and other gases. As reproducible data have not yet been obtained in these calibration runs, it is believed that adsorption of nitromethane on the walls is an inherent difficulty in the analysis. As adsorption is dependent on pressure, temperature, time of contact, and the presence of other gases, it may very easily account for some of the inconsistencies observed.
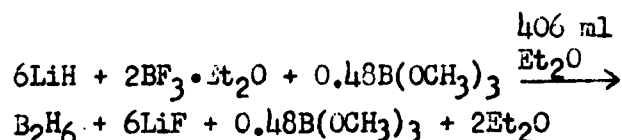
2. The mass spectrometer is also employed on a number of other projects, and therefore cannot always be used for work under this contract. For this reason, the technique necessary for accurate analysis has not yet been worked out, although it is believed that the problem will be solved in the very near future.

IV. PILOT PLANT PREPARATION OF LITHIUM BOROHYDRIDE IN HYDRAZINE

A. Pilot plant preparation of a 13% solution of lithium borohydride in hydrazine has been completed, a total of 27.6 lb having been made in 12 production runs. The details of the method of production have been described previously in References 1 and 16.

B. The reactions employed for the preparation of lithium borohydride, together with the average quantities of the reactants and products, are shown below:

Theoretical reaction:

$$6LiH + 2BF_3 \cdot Et_2O + 0.48B(OCH_3)_3 \xrightarrow[Et_2O]{406 \text{ ml}}$$
$$B_2H_6 + 6LiF + 0.48B(OCH_3)_3 + 2Et_2O$$

Experimental reaction: (moles)

$$6.00 \qquad 4.08 \qquad 0.505$$

Theoretical reaction:

$$B_2H_6 + 2LiH \xrightarrow[Et_2O]{1310 \text{ ml}} 2LiBH_4$$
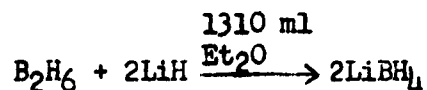
Experimental reaction:

$$* \qquad 2.00 \qquad 1.41**$$

* Not measured. Diborane generated is fed directly into lithium borohydride generator.
** Average of Runs 1-12.

Complete information on each of the production runs is given in Table I.

IV Pilot Plant Preparation of   Report No. 82.-26
   Lithium Borohydride in Hydrazine, B (cont.)

       1.   Following Run No. 7, it was determined that the lithium hydride purity was only 91.7%, in contrast with the manufacturer's asserted average value of 98%. All data reported in Table I have been corrected to the actual active hydride content of the starting material. Yields are reported in Table I on the basis of the lithium hydride used in step 2 of the synthesis, and also in terms of the total lithium hydride used (steps 1 and 2). The low over-all yields result from the fact that considerably more diborane was prepared in step 1 than could be absorbed in step 2. Marked improvement in the over-all yield undoubtedly could be achieved by a determination of the optimum amount of lithium hydride required in step 1.

       2.   Because many of the production runs resulted in lithium borohydride purities of only 80 to 90%, it was suspected that the major portion of the remainder of the sample might be residual ether that had not been removed by heating the sample to $60^{\circ}C$. Accordingly, after Run No. 8, samples to be analyzed were heated to between 80 and $100^{\circ}C$ for 20 min, while being evacuated to a few mm Hg pressure; an increase in purity of approximately 10% has resulted from this operation.

       3.   Run No. 12 was performed in the same manner as the previous runs, with the exception that boron fluoride etherate was used that had not been redistilled. The high yield indicated that it probably is not necessary to employ freshly distilled material for satisfactory production of diborane.

       4.   It was noticed that after the various batches of lithium borohydride were dissolved in hydrazine, a material was suspended throughout the entire solution. This gray suspension, which amounted to less than 0.1% of the total quantity of fuel, eventually settles to the bottom of the container and is then removed in the filtration operation. A spectrographic analysis of the washed substance showed the presence of 14% lithium and 7.8% boron as the chief metallic constituents.

       5.   For a series of motor tests, several individual preparations, each calculated to contain 14% lithium borohydride, were accumulated in the blending vessel, from where they were pumped through a filter into the portable fuel test tank. Additional hydrazine was then added, as shown necessary by an analysis of the composite solution, to reduce the strength to approximately 13%. The last columns of Table I show the details of the quantities involved in this step.

      C.   Although the analysis of material from the pilot plant is not difficult, the techniques employed are nevertheless specialized enough to warrant a brief outline of the methods of determination of (1) total solids, (2) lithium borohydride, and (3) lithium borohydride in the final blended fuel.

       1.   All glassware in which samples are collected or weighed is dried at $110^{\circ}C$, flushed with nitrogen, and tightly stoppered.

IV Pilot Plant Preparation of   Report No. 820-28
    Lithium Borohydride in Hydrazine, B (cont.)

        2.   To determine the total solids in the filtered ether solution, approximately 35 g of the solution is pressurized from the filter discharge and accurately weighed. A three-way stopcock (one arm to the flask, one to a dry nitrogen supply, and the third vented to the atmosphere) is adjusted so that ether may evaporate from the solution while a slow stream of nitrogen protects the sample from absorbing moisture. The flask is slowly heated to 80°C, at which temperature only a small quantity of liquid remains. Vacuum is then applied to the vent arm (the nitrogen having been turned off) and the flask is heated at 80 to 100°C for 20 min. An atmosphere of nitrogen is introduced into the evacuated flask, and the flask is cooled, dried, and reweighed in order to determine total solids.

        3.   Two samples of approximately 0.1 g of the dried lithium borohydride are placed in tared, 25-ml Erlenmeyer flasks in a nitrogen-filled dry box, and the weight of sample accurately determined. A nitrogen-flushed, 1000-ml eudiometer is adjusted to atmospheric pressure with the sample flask in place, the leveling bulb is then lowered to produce a subatmospheric pressure within the eudiometer to check for leaks and to prevent a positive pressure when hydrogen is released from the sample, and one drop of water from the burette is allowed to fall on the lithium borohydride. If the eudiometer is not flushed with nitrogen, a small flame often results from this first addition and causes a serious error in the results. Water is now slowly added (the flask being continuously cooled by a water bath) until the reaction has subsided; water amounting to a total of 6 ml is added. The further addition of 6 ml of 6N hydrochloric acid ensures complete hydrolysis of the sample.

        4.   The recorded gas volume is corrected for the volume of liquid added from the burette (12 ml), and corrected to 0°C and 760 mm Hg pressure. The purity of the sample is then calculated from

$$\frac{\text{Corrected gas volume}}{\text{Weight of sample}} \times \frac{1}{4114} \times 100 = \% \text{ purity } LiBH_4$$

        5.   In the determination of lithium borohydride concentration in the hydrazine solution, samples of approximately 0.7 g are accurately weighed in 25-ml Erlenmeyer flasks and the volume of hydrogen released on hydrolysis measured in the eudiometer. Three ml of water and 9 ml of 6N acid are used with these samples, and the sample flask is heated to 80°C to ensure that all of the hydrogen is evolved. The solution is allowed to return to room temperature before the gas volume reading is taken.

V.   ENGINE TEST PROGRAM

A.   HYDRAZINE-OXYGEN CALIBRATION TESTS

A series of performance tests of pure hydrazine were made with liquid oxygen in order to establish a basis for evaluation of the performance of the solution of 13% lithium borohydride in hydrazine. These tests have been previously reported (Reference 1), together with a description of the 100-lb-thrust engine used.

B.   13% LITHIUM BOROHYDRIDE IN HYDRAZINE-OXYGEN TESTS

1.   Fourteen tests were made with the solution of lithium borohydride in hydrazine; a summary of performance data is given in Table II. Although one specific impulse as high as 262 lb-sec/lb was noted, a smoothed curve through the data indicates a maximum of approximately 256 lb-sec/lb. This latter value is 94% of the maximum theoretical specific impulse. Plots of mixture ratio vs specific impulse, characteristic exhaust velocity, and thrust coefficient are shown in Figure 14.

2.   The injector used for the tests of the lithium borohydride solution was that which had been used with the hydrazine calibration tests (Figure 11). It had 10 pairs of 1:1 impinging holes. The resultant momentum is axial at a mixture ratio, w_o/w_f, of 0.87, and the point of impingement is 1/16 in. from the face of the injector.

3.   All runs were characterized by copious deposits of gray or white oxides of varying hardness, which almost completely covered the chamber and nozzle walls as well as most of the turbulator and injector face. The injector holes were seldom covered, however, and no injector burnouts occurred. Figure 12, made after Test No. 14 shows, at the left, the injector face and at the right, the upstream side of the chamber and turbulator. Figure 13, photographed after Test No. 14, shows, at the left, the upstream side of the nozzle and at the right, the downstream end of the chamber. Part of the nozzle coating is not shown, having broken away in disassembling the engine. In spite of the heavy deposits, all tests were characterized by smooth performance. Injection, chamber pressure, and thrust were generally constant for an adequate test interval (5 sec). Tests No. 11 to 15 were performed with a stainless steel chamber liner with the intention of reducing to a minimum the heat-transfer correction. However, because of severe progressive erosion of the liner near the outer turn of the turbulator, it was necessary to complete the testing with a copper liner. Erosion of the copper liner was slight. There was little if any damage to the copper turbulator in any of the tests. Heat-transfer corrections ranged from 3.8 to 8.8%, with an average of 5.6% when the stainless steel liner was used. The corresponding value using the copper liner were 4.9 to 6.7%, with an average of 5.6%. Obviously, use of the stainless steel liner did not result in smaller heat-transfer corrections.

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TABLE I

PILOT PLANT PREPARATION OF LITHIUM BOROHYDRIDE IN HYDRAZINE

(Step 1: Preparation of Diborane)

Run No.	LiH[1] mole	Ether liters	$B(OCH_3)_3$ mole	$BF_3 \cdot Et_2O$ mole	Time[3] hr
1	23.9	3.28	–	14.9	3.25
2	30.9	1.75	2.78	18.68	1.83
3	30.9	1.75	2.78	18.68	1.75
4	51.4	2.91	4.61	31.0	4.00
5	69.3	4.0	6.0	52.7	7.00
6	69.3	4.0	6.0	46.2	7.00
7	69.3	4.0	6.0	57.4	8.00
8	69.3	4.0	6.0	46.2	6.75
9	69.3	4.5	6.0	46.2	6.50
10	69.3	5.5	6.0	46.2	6.75
11	69.3	5.5	6.0	46.2	6.00
12	69.3	5.5	6.0	46.2[2]	6.00
13	69.3	5.5	6.0	46.2[2]	5.16

(Step 2: Preparation of Lithium Borohydride)

Run No.	LiH mole	Ether liters	Purity $LiBH_4$, %	Weight 100% $LiBH_4$, g	Yield[4] %	Over-all Yield[6] %
1	4.51	4.2	38.2	–	–	–
2	9.07	6.0	99.4	187.5	63.3[5]	77.4
3	9.07	6.0	83.8	85.5	43.5	39.2
4	15.10	10.0	94.5	235.5	72.2	65.0
5	15.10	10.0	91.6	334.0	100.2	72.5
6	15.0	10.0	80.5	255.0	78.0	55.4
7	15.0	10.7	83.5	178.0	54.8	38.7
8	15.0	10.7	84.5	138.0	42.3	30.0
9	15.0	11.0	95.0	214.0	65.5	46.5
10	15.0	11.0	92.5	283.0	86.7	61.5
11	15.0	11.0	93.2	221.0	67.7	48.0
12	15.0	11.0	94.7	290.0	88.9	63.0
13	15.0	11.0	95.2	325.0	99.5	71.0

[1] Calculated from amount of 91.7% pure LiH used.

[2] Undistilled.

[3] Total time for Steps 1 and 2.

[4] Based on 100% LiH and 100% $LiBH_4$ in Step 2.

[5] Based on LiH used in Step 2 of Runs 1 and 2. A yield may be favorably affected by unreacted LiH of previous run.

[6] Based on total 100% LiH, Steps 1 and 2.

Table I
Sheet 1 of 2

CONFIDENTIAL

TABLE I, STEP 2 (cont.)

Run No.	Wt as 100% LiBH$_4$ g	Wt as 100% LiBH$_4$ Mole	Wt N$_2$H$_4$, g per run	Wt N$_2$H$_4$, g added in blending	LiBH$_4$ in N$_2$H$_4$ % Calc	LiBH$_4$ in N$_2$H$_4$ % by anal.	Motor Tests
1	-	-	-	-	-	-	-
2	187.5	8.59	1200	↑	↑	↑	↑
3	85.5	3.92	655				
4	235.5	10.79	1226	312	13.6	13.06	11-15
5	334.0	15.30	2040				
6	255.0	11.69	1635				
7	178.0	8.16	1041	↓	↓	↓	↓
8	138.0	6.32	847	↑	↑	↑	↑
9	214.0	9.81	1542	563	12.4	12.3	16-20
10	283.0	12.97	1739	↓	↓	↓	↓
11	221.0	10.13	1357				
12	290.0	13.29	1677	113	13.0	13.2	21-24
13	325.0	14.94	2130	113	13.0	13.2	21-24

Table I
Sheet 2 of 2

TABLE II

LITHIUM BOROHYDRIDE IN HYDRAZINE AND OXYGEN

Summary of Performance Data

Test No. 37 L_F	Fuel	Oxidizer	Chamber Press., psia	M.R. w_{ox}/w_f	C_F (corr. for heat transfer)	I_{sp}, lb-sec/lb (uncorrected)	c^*, ft/sec (corr. for heat trans.)	I_{sp}, lb-sec/lb (corr. for heat transfer)
11	12.34% LiBH₄ in N₂H₄	Liquid Oxygen	297	0.96	1.41	240	5676	249
12			300	0.80	1.39	231	5683	245
13			304	1.07	1.38	233	5806	248
14			293	0.96	1.40	245	5880	256
15			284	0.99	1.42	237	5689	250
16			308	1.12	1.40	240	5769	252
17			310	0.93	1.41	239	5810	255
18	13.06% LiBH₄ in N₂H₄		323	1.06	1.40	246	5945	259
19			317	1.14	1.40	247	6025	262
20			303	0.80	1.39	239	5915	256
21			321	1.28	1.42	247	5860	258
22			299	1.42	1.42	243	5800	255
23			308	1.25	1.38	243	5985	256
24			305	1.38	1.39	244	5940	256

Table II

METHYLHYDRAZINE—HYDRAZINE

FREEZING POINT DIAGRAM

WEIGHT PER CENT METHYLHYDRAZINE
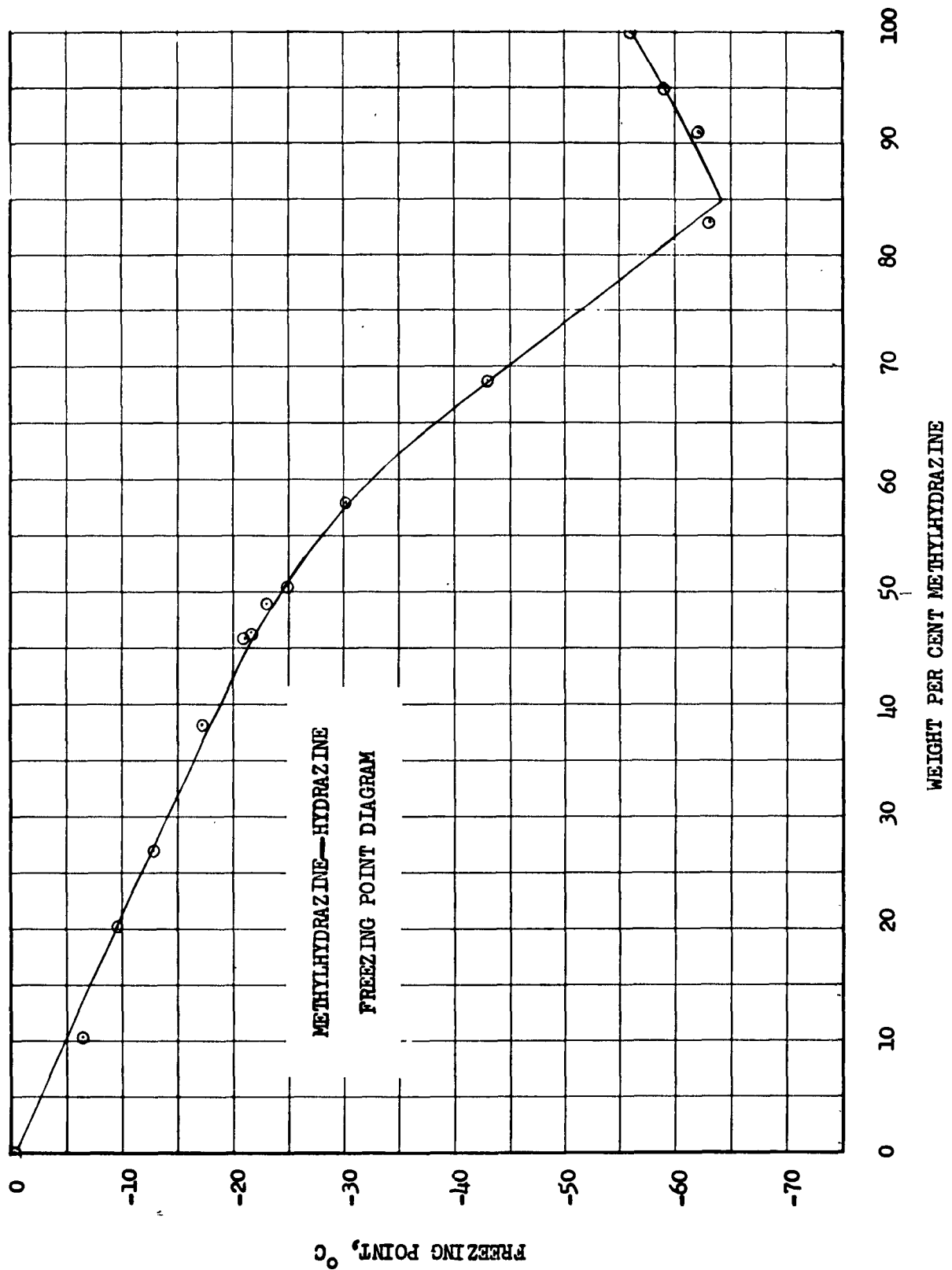
FREEZING POINT, °C

Figure 1

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15 July 1952   Progress Report No. 820-28

40

INVESTIGATION OF

LIQUID ROCKET PROPELLANTS

Contract N7onr-462
Task Order III
Project No. NR 220 023

ERRATUM SHEET

In Figure 2, METHYLHYDRAZINE-HYDRAZINE MOLE PER CENT VS. FREEZING
POINT, the ordinate scale should be displaced downward by 5°.

Approved by: *D. L. Armstrong*
D. L. Armstrong
Principal Chemist
Solid Engine and
Chemical Division

AEROJET ENGINEERING CORPORATION

Azusa, California

METHYLHYDRAZINE—HYDRAZINE

Mole Per Cent vs. Freezing Point

THEORETICAL CURVE

MOLE PER CENT METHYLHYDRAZINE

FREEZING POINT, °C

Figure 2

MONOMETHYLHYDRAZINE—LIQUID OXYGEN

SPECIFIC IMPULSE

VS.

MIXTURE RATIO

Figure 3

G-3202   CBE   3-25-52

N, N-DIMETHYLHYDRAZINE—LIQUID OXYGEN

SPECIFIC IMPULSE

VS.

MIXTURE RATIO

Specific Impulse, lb-sec/lb

Mixture Ratio

Figure 4

MONOMETHYLHYDRAZINE—LIQUID OXYGEN

CHAMBER TEMPERATURE, THRUST COEFFICIENT,

CHARACTERISTIC VELOCITY, AND MEAN HEAT

CAPACITY RATIO VS. MIXTURE RATIO

C-3200   CBE   3-25-52

Chamber Temperature, °K

Characteristic Velocity, ft/sec

Mean Heat Capacity Ratio

Thrust Coefficient

⊙ c*
X T_c
+ k
△ c_F

6300

6200

6100

6000

5900

5800

3400

3300

3200

3100

1.20

1.15

1.10

1.42

1.41

1.40

1.39

Mixture Ratio

1.00   1.10   1.20   1.30   1.40   1.50   1.60   1.70   1.75

Figure 5

Report No. 820-28

N, N-DIMETHYLHYDRAZINE—LIQUID OXYGEN

CHAMBER TEMPERATURE, THRUST COEFFICIENT, CHARACTERISTIC VELOCITY, AND MEAN HEAT CAPACITY RATIO VS. MIXTURE RATIO

Mixture Ratio

Figure 6

REMOTE CONTROL HANDLE

ELECTROMAX

VARIABLE RESISTANCES

POTENTIOMETER

S-10328

HYDRAULIC JACK

AMPOULE HOLDER EXTENSION

SAFETY SHIELD

FURNACE

QUENCHING BATH

MIRROR

THERMAL DECOMPOSITION FURNACE ASSEMBLY

S-10328

C

MUELLER

AIRCRAFT ENGINEERING CORP.
AZUSA, CALIFORNIA

4-8-52

MANNING

1/8

SCALE

Report No. 820-28

0452-331

Ampoule and Ampoule Holder

Figure 8

Figure 9

Report No. 820-28

TO POTENTIOMETER
(CHECK)

TO POTENTIOMETER

THERMOCOUPLE

THERMOCOUPLE (CHECK)

PLUG

AMPOULE HOLDER

FURNACE
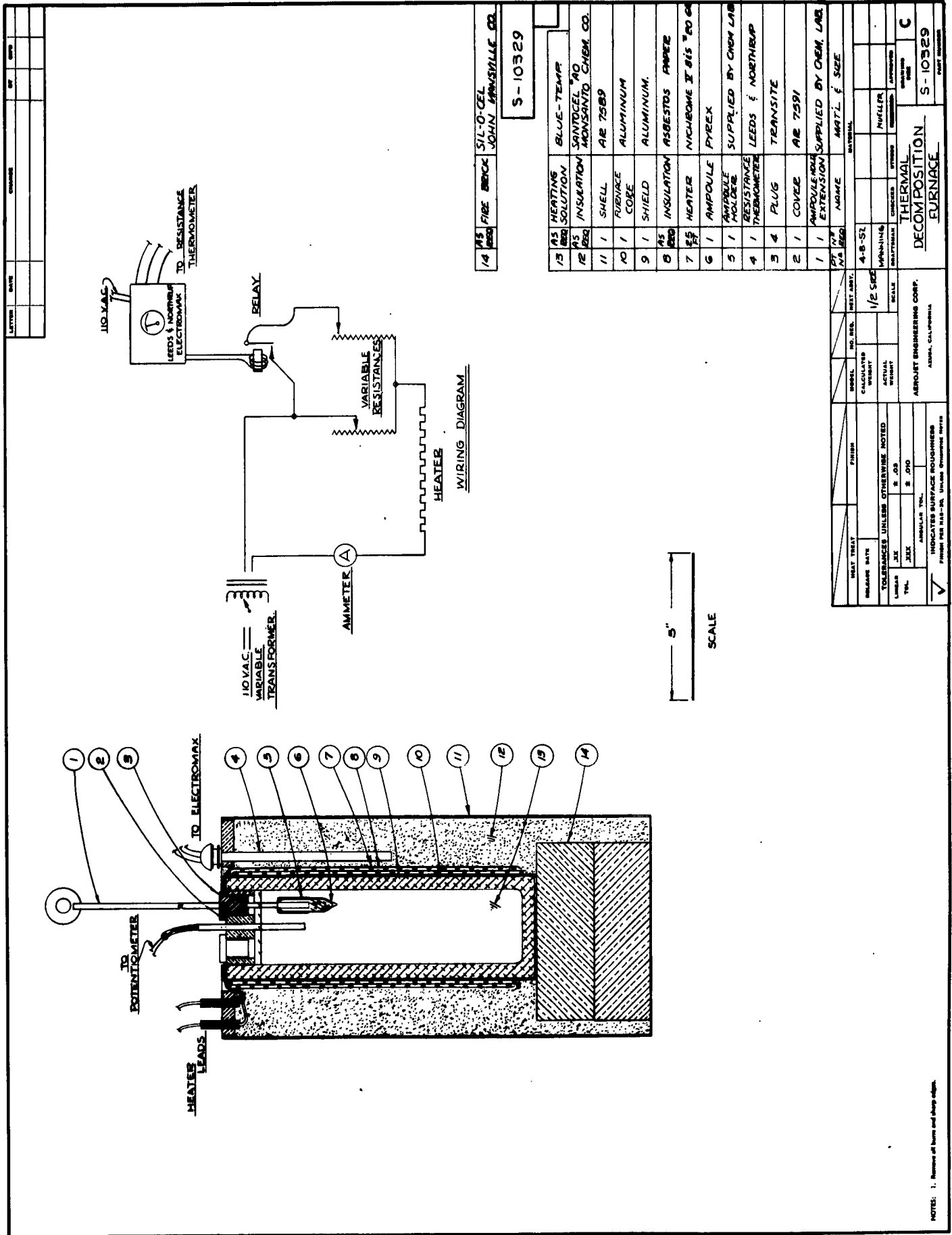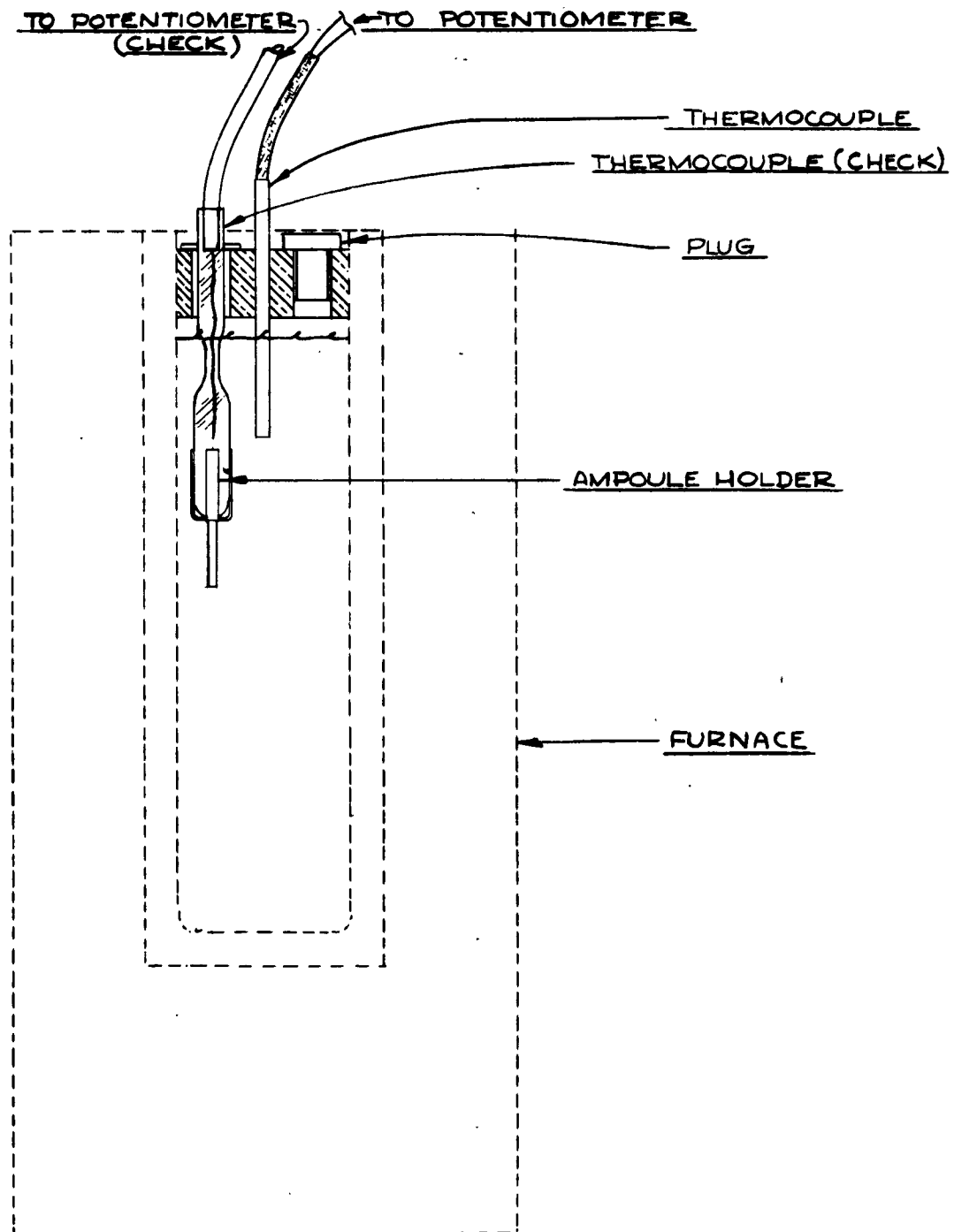
MODEL	NO. REQ.	NEXT ASSY.	MATERIAL					
CALCULATED WEIGHT		½ SIZE	4-8-52					
ACTUAL WEIGHT			MANNING			MUELLER		
		SCALE	DRAFTSMAN	CHECKED	STRESS		APPROVED	

AEROJET ENGINEERING CORP. AZUSA, CALIFORNIA	TEMPERATURE CHECK ASSEMBLY DECOMP. FURNACE	DRAWING SIZE	C
		S-10330	
		PART NUMBER	

Figure 10

ORIFICE PLATE BLANK
SCALE~ 2 X SIZE

1.060 DIA.
.820 DIA.
.800, .805 DIA.
.555 .350 DIA.
.410 .400
.200
29°
41°
20°
.500 NOM.
DIA.:UP FILLET (2)
.06 R.

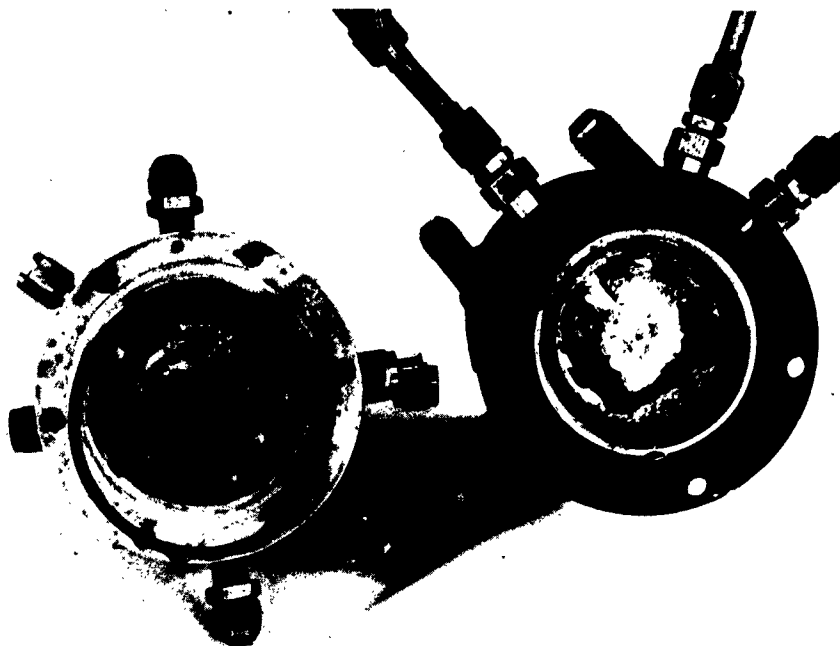SECTION B-B

DRILL #72 (.025) THRU
10 HOLES EQ SPACED WITHIN .005
LOCATE FROM IMPINGMENT POINT

IMPINGMENT POINT

#3 OF THE 2 HOLES MUST
INTERSECT WITHIN .005
(IMPINGMENT POINT)
TYP. 10 PLACES

DRILL #70 (.028) X .128 DEEP
10 HOLES EQ SPACED WITHIN .005
LOCATE FROM IMPINGMENT POINT

.276 R
.271 R
29°
41°

(3) PLATE - ORIFICE - 1 REQ.
MAT: ELECTROLYTIC COPPER
SCALE~ 5 X SIZE

SCRIBE LINE ON OD
WITHIN .004 OF THIS
℄ (RELATION TO HOLES)

36° TYP

DRILL .062 DIA. THRU ON
5° ANGLE. 5 HOLES EQ. SPACED
BETWEEN EXISTING HOLES
10 HOLES MUST BE EQ. SPACED
WITHIN .005

.742 H.C. REF.

36° TYP.

SECTION A-A

NOTE: THIS VIEW FOR REWORK OF (2)

SILVER BRAZE (USE .005 FOIL)
JOINT MUST BE PERFECT OVER
ENTIRE SURFACE. SCRIBED
LINES MUST BE IN LINE
WITHIN .002
PRESSURE TEST AT 500 P.S.I.

.998 .996 DIA.

40°

5° TYP.

DRILL .062 DIA. X .265 DEEP
10 HOLES IN (3) LOCATE
FROM (2) AFTER BRAZING

6-.998 .996 DIA. REF.

ASSEMBLY

NOTES: 1. Remove all burrs and sharp edges.

ALTERNATE FOR AR 20028-3

USE WITH INJECTOR AR 20045

PT. NO.	NO. REQ.	NAME	MATERIAL & SIZE
3	1	PLATE ORIFICE	ELECTROLYTIC COPPER 1.125 DIA. X .500 LG.
2	1	CORE	AR 20028-2 (REWORK)
1	1	TUBE	AR 20028-1

HUGHES	12-5-51	DRAFTSMAN
P. HAHN	12-7-51	CHECKED
R. Sandgren	12-7-51	STRESS ENGINEER
		APPROVED

AEROJET ENGINEERING CORP.
AZUSA, CALIFORNIA

TOLERANCES UNLESS OTHERWISE NOTED
LINEAR .XX ±.03
.XXX ±.010
ANGULAR TOL. ±0°30'

INJECTOR CORE
1:1 IMPINGING
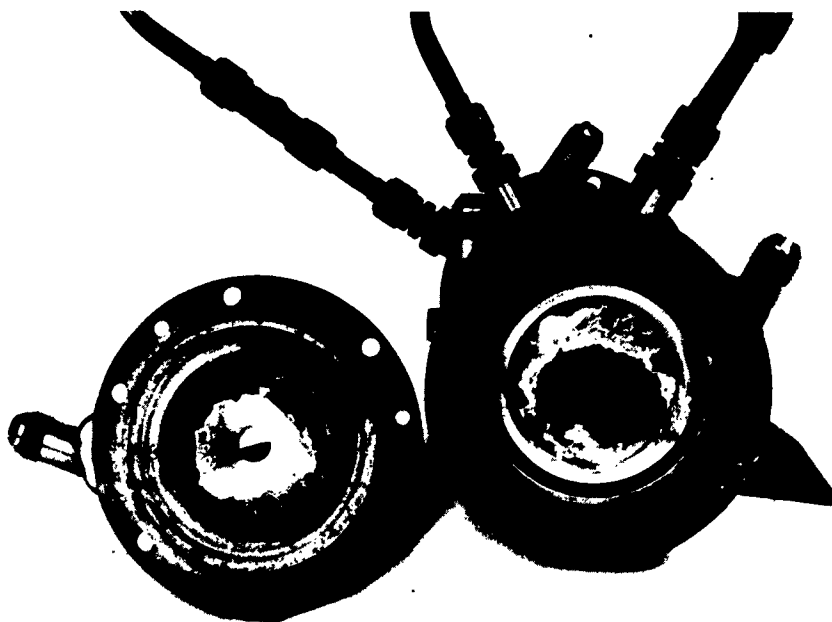ALTERNATE FOR AR20028

SIZE	D	AR 7659

AR 7659

Figure 11

0352-565

Fig. 12 Injector Face and Chamber

0352-564

Fig. 13 Nozzle and Nozzle End of Chamber

Figures 12 and 13
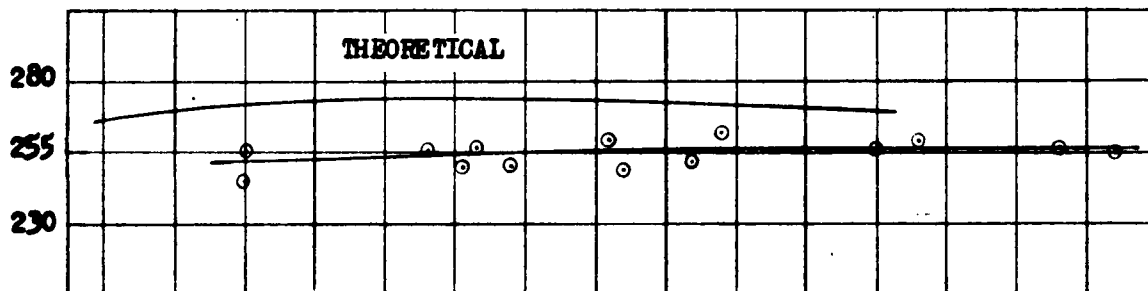
PERFORMANCE VERSUS MIXTURE RATIO
FOR 13% LITHIUM BOROHYDRIDE IN HYDRAZINE
AND LIQUID OXYGEN

DATA CORRECTED FOR HEAT TRANSFER
F ≈ 100 POUNDS
P_c ≈ 300 psia
TEST NOS. D37LF 11-24
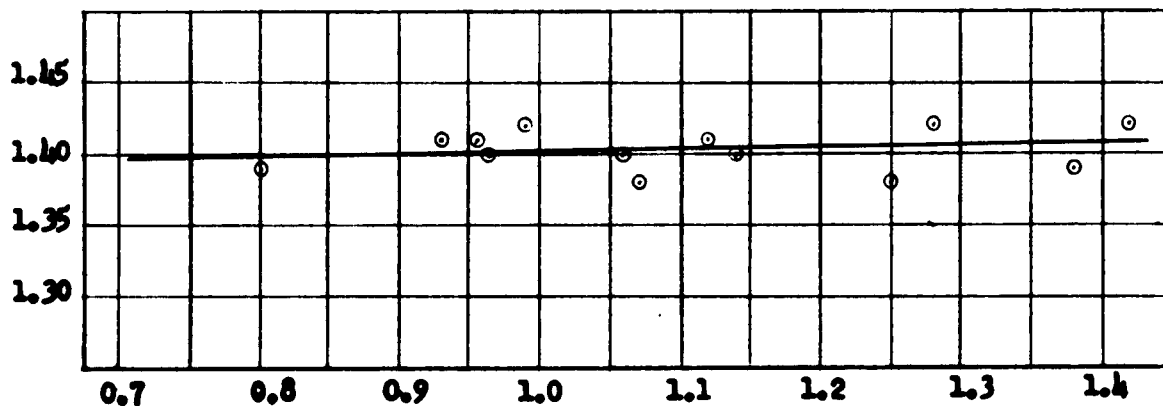

Figure 14

C-3220 ESM/ab 5-6-52